

**ADVANCED AIR-BREATHING ENGINE TECHNOLOGY —
MATERIALS: A REVIEW OF CONTRACTS**

by S. J. Grisaffe

**Lewis Research Center
Cleveland, Ohio**

**TECHNICAL PAPER proposed for presentation at
Eleventh Meeting of the Refractory Composites Working Group
Los Angeles, California, January 24-28, 1966**

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

ADVANCED AIR-BREATHING ENGINE TECHNOLOGY —

MATERIALS: A REVIEW OF CONTRACTS

by S. J. Grisaffe

Lewis Research Center
National Aeronautics and Space Administration
Cleveland, Ohio

E-3328

The NASA Lewis Research Center is deeply involved in the development of advanced air-breathing jet engines. A portion of the Lewis effort involves contractual programs focused on one of the most pressing problems in such engines — materials for hot section components. The following is a description of those programs which have been contracted during the last year with research teams across the country to advance the technology for higher temperature engine materials. The program augments existing Air Force, Navy, and Army programs in this general area. Program management is under the supervision of the Lewis Spacecraft Technology Division with technical guidance being supplied by the Lewis Materials and Structures Division.

CHROMIUM

With a melting temperature of 3430° F, a relatively low density, and good thermal conductivity, chromium offers intriguing possibilities as a relatively oxidation-resistant alloy base for use in the 2000° to 2500° F range. Chromium, however, also has drawbacks, the most serious of which are its high ductile-to-brittle transition temperature and its tendency toward interstitial embrittlement, particularly by nitrogen. A concerted effort is being expended to develop promising chromium systems using both melting and powder metallurgy techniques. A description of the contractual programs dealing with this overall effort is now presented.

Forgeable Chromium-Base Alloys (NAS 3-7260)

General Electric Co., Evendale, Ohio)

Chromium alloys are being prepared primarily by induction melting and vacuum-arc melting techniques. A wide range of compositions, involving both solid solution strengthening and carbide precipitation strengthening, will be evaluated for high-temperature tensile strength, oxidation resistance, and ductile-brittle transition temperature, and will also be analyzed by microstructural studies after various heat treatments. In addition, the more promising of these alloys will be subjected to high-temperature creep tests. Based on these results a few selected alloys will be further characterized for fabricability and allied factors.

Dispersion-Strengthened Chromium Alloys

The following contracts are aimed at producing very fine oxide dispersions in a chromium matrix with subsequent consolidation being accomplished by powder metallurgy techniques.

Vapor plate (NAS 3-7608 Melpar, Inc., Falls Church, Va.). - Composite powders will be produced by the vapor phase deposition of chromium onto oxide particles in a fluidized bed reactor. The resultant composite powders with several volume percentages of oxide will be extruded and fabricated into test specimens. In this program both MgO and ThO₂ will be studied as dispersoids in the unalloyed chromium matrix.

Ball mill (NAS 3-7607 General Electric, Evendale, Ohio). - A complex alloy (Cr-Mo-Y-Th-Hf) will be dispersion strengthened with several volume percentages of yttria or magnesia. Materials will be prepared by ball milling in an inert atmosphere followed by consolidation. The consolidated

alloy will be tested for stability of dispersoid, oxidation/nitridation characteristics, ductile-to-brittle transition temperature, and 2000° F tensile strength.

Electrodeposition (NAS 3-7606 General Technology Corp., Alexandria, Virginia). - Unalloyed chromium will be deposited from aqueous electrolytic plating baths containing selected dispersoids. The dispersoids to be studied will be alumina, magnesia, and thoria. After deposition, the materials will be crushed and consolidated by one of several standard techniques.

Chromium Alloy Coating Programs

Regardless of the consolidation route followed, it is anticipated that chromium alloys will need to be coated to improve oxidation resistance and to protect against embrittlement by nitrogen. The following coating contracts utilize a Cr-5W-0.05 Y alloy substrate (NAS 3-7901, General Electric, Cleveland, Ohio); any further work would be expected to use a stronger more ductile alloy if such becomes available.

Aluminide coatings (NAS 3-7273 Chromalloy Corp., W. Nyack, N.Y.). - Pack cementation aluminide type coatings will be evaluated in static-cyclic oxidation for times up to 600 hours at temperatures up to 2400° F. Such tests will be supplemented by bend transition temperature studies as well as burner rig oxidation-erosion experiments.

Silicide coatings (NAS 3-7266 Solar, San Diego, Calif.). - Two approaches are to be taken in this program. One involves the deposition of a diffusion barrier (Re) covered with a titanium-chromium alloy which will be partially silicided. The second approach involves a vanadium containing alloy layer

to act as an interstitial sink for nitrogen. This layer will then be partially silicided for maximum oxidation protection. Static-cyclic oxidation and bend transition tests will be conducted. Oxidation exposures of up to 600 hours will include a low temperature exposure in the pest region as well as elevated temperature exposures of up to 2400° F. Impact tests will also be conducted on these systems.

Metallic claddings (NAS 3-761 Battelle Memorial Institute, Columbus, Ohio). - Similar tests as described previously will be conducted on metallurgically clad specimens which have been processed by isostatic hot pressing. Inner metallic diffusion barriers as well as outer oxidation-resistant nickel-chromium alloys will be applied by this technique. In some instances the oxidation resistance afforded by aluminizing the surface alloys will also be studied. Again, oxidation exposures will be for times up to 600 hours at temperatures up to 2400° F.

NICKEL AND COBALT-BASE SUPERALLOYS

The most reliable materials employed to date in commercial and military jet engine hot sections have been the superalloys. These materials will be the mainstay of engine manufacturers for the immediate future and further advances in these material systems are still possible. The following programs have been designed to explore such advances by several different manufacturing techniques.

Cobalt-Base Alloys (NAS 3-7600 TRW, Cleveland, Ohio)

A variety of alloy compositions will be studied in a statistically designed manner to attempt to develop a precision cast cobalt-base alloy having a 3000-hour stress rupture life at 2125° F under a 4000-psi stress;

this alloy is not to be subject to catastrophic oxidation at 2125° F, but it is expected that a protective coating will be required for long-time operation at this temperature. Furthermore, this alloy should be resistant to thermal cycling and have a degree of workability

Nickel-Base Alloys (NAS 3-7267 TRW, Cleveland, Ohio)

A similar program is devoted to the development of a precision cast nickel-base alloy having a 3000-hour stress rupture life at 1875° F under a 15 000-psi stress. This alloy should not be subject to catastrophic oxidation at 1875° F, should be resistant to thermal cycling, and should have some degree of workability.

Dispersion-Strengthened Superalloys

Arc process (NAS 3-7275 Vitro Laboratories, W. Orange, N.J.). - A solid solution nickel alloy matrix containing tungsten, cobalt, and molybdenum, plus a dispersoid of thoria will be prepared by electrode vaporization in a high-intensity arc. Following this step, the resultant powders will be reduced in hydrogen and consolidated.

Vapor deposition using fluidized bed (NAS 3-7271 Melpar, Inc., Falls Church, Va.). - A fluidized bed will be used to vapor deposit layers of nickel, chromium, and molybdenum onto submicron thoria and zirconia powders. The volume of the dispersoids in the alloy matrix will be varied up to 8 percent. Selected billets will be extruded, and the subsequent materials systems tested.

Stabilize precipitate plus mechanical mixing (NAS 3-7279 Ilikon Corp., Natick, Mass.). - The intent of this program is to develop a solid solution nickel-base alloy strengthened by internal oxidation. After the alloy is

produced, the gamma prime precipitate and internal oxidation products are developed. The final steps are stabilization and deoxidation.

Salt precipitation and selective reduction to form prealloyed powder (NAS 3-7272 Curtiss-Wright, Buffalo, N.Y.). - A cobalt alloy, solid solution strengthened with lanthana or thoria, is to be prepared by formation and reduction of metallic salts. This material will then be consolidated by standard techniques.

Selective reduction plus mechanical mixing (NAS 3-7265 New England Materials, Medford, Mass.). - The approach to be taken in this study is one of selective reduction of mixed oxides to produce a dispersion-strengthened solid solution alloy powder (nickel base) which will then be processed by standard powder metallurgy techniques.

Salt precipitation and selective reduction (NAS 3-7611 Sylvania, Towanda, Pa.). - An oxidation-resistant cobalt-base alloy containing nickel, chromium, and molybdenum is to be prepared with several percentages of either calcia or thoria additions. Salts of the constituents are to be selectively reduced to form the desired matrix containing the dispersed oxides.

Superalloy powder purification (NAS 3-7274 Sylvania, Towanda, Pa.). - This program requires that a feasibility apparatus be constructed whose purpose is to hydrogen reduce submicron nickel powders during "fluidization with transport". The goal is 100 grams per 8-hour day of 0.15-micron powder with a maximum oxygen content of 0.05 percent. The possibilities for scaleup will be considered.

Fiber Metallurgy

Stable refractory oxide fibers (NAS 3-7903 Monsanto, Everett, Mass.). -

A study is being conducted to determine the required conditions whereby oxide fibers may be drawn in a manner similar to glass. Alumina and two other oxides are to be studied and the resultant fibers are to be examined optically, by x-ray diffraction, and by room temperature and hot tensile tests after a variety of annealing treatments.

Refractory metal alloy fibers (NAS 3-7906 General Electric, Cleveland, Ohio). - Several of the stronger molybdenum, columbium, and tungsten alloys are being studied for drawing into wire less than 10 mils in diameter. The ultimate objective for this study is to provide high strength, refractory metal wires for incorporation into a metallic matrix to form a fiber-reinforced composite. Stress rupture and tensile tests will be conducted on the wires at temperatures up to 2400° F for times up to 1000 hours.

COATINGS FOR TANTALUM

Although coated refractory metals do not appear promising for long-time service in rotating machinery of advanced air-breathing engines, there does seem to be some opportunity for their use in nonrotating components. Tantalum alloys appear to have the required strength for such operation in the 2000° to 2500° F range. Tantalum has the highest melting-point oxide of the four common refractory metals (W, Mo, Cb, and Ta), and it has received comparatively little previous study for service below 2500° F. In order to explore the possibility of protecting a tantalum-base alloy for use in air at temperatures below 2500° F, the following programs, involving coating of the alloy T-222 (Ta-10.6W-0.4Hf-0.01C), have been initiated.

Silicide Coatings on Diffusion Barriers

(NAS 3-7276 Solar, San Diego, Calif.)

A number of duplex silicide coatings will be deposited on metallic diffusion barriers and will be evaluated in static-cyclic oxidation at temperatures up to 2400° F for times up to 600 hours. Bend transition temperature and impact tests will also be conducted in an attempt to determine if surface alloying with subsequent siliciding offers a potential protection scheme for refractory metal stator vane material.

Miscellaneous Coatings on Diffusion Barriers

(NAS 3-7613 Vitro Laboratories, W. Orange, N.J.)

Refractory metal diffusion barriers are being deposited electrophoretically and sintered. This treatment is being followed by the electrophoretic deposition of a number of potentially oxidation-resistant intermetallic compounds which are also sintered. After deposition parameters are optimized, these systems will be tested in static-cyclic oxidation for times up to 600 hours at temperatures up to 2500° F. Again the substrate of interest is the tantalum alloy T-222.

MISCELLANEOUS

Oxidation Resistant Materials for Wires - Transpiration-Cooled

Components (NAS 3-7269 Bendix Filter Div., Detroit, Mich.)

Commercial iron, nickel, and cobalt-base superalloys are being studied for potential use in "Porolloy" type materials. Preliminary oxidation studies of sheet and wire specimens are being conducted for times up to 600 hours at temperatures to 2200° F by static furnace tests.

Prediction of Cooled Turbine Blade Metal Temperatures

(NAS 3-7278 General Electric Co., Evendale, Ohio)

An analytical study is being conducted to predict metal temperature distributions on edge-cooled, convection-cooled, and transpiration-cooled blades.

Steady-State Creep of Dispersion-Strengthened Metals

(NAS 3-7615 Battelle Memorial Institute, Columbus, Ohio)

The creep behavior of fine-grain, dispersion-strengthened nickel is being studied in order to define the nature of the interactions between dislocations and dispersions and to relate the particle size and spacing effects to the theories of Ansell and Weertman. Twenty-mil sheet stock of thoriated nickel has been prepared by Sherritt-Gordon with thoria loadings of 1, 3, and 5 percent and with two levels of thoria particle size (150 to 300 Å and 500 to 800 Å). Creep tests and transmission electron microscopy are being carried out.